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MAY 79 C G HOMAN, D P KENDALL, R K MACCRONE

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C. G. Homan
D. P. Kendall
R. K. MacCrone

May 1979

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER WEAPON SYSTEMS LABORATORY
BENÉT WEAPONS LABORATORY
WATERVLIET, N. Y. 12189

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Magnetic moment measurements of cadmium sulfide (CdS) polycrystalline samples, pressure quenched from above the semi-conducting to conducting transition pressure (40 kbars) at rates approaching 10^6 bars/sec, have been made in a vibrating sample magnetometer to fields approaching 1000 Oersted. The samples exhibit the complex magnetic behavior of low field diamagnetism ($\chi_v > -5\times10^{-5}$ cgs units) transforming at fields of several hundred Oersted to positive magnetism.

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to positive magnetism ($\chi_V > 3\times10^{4}$ cgs units). This magnetic behavior contrasts with unquenched samples which yield the normal diamagnetism of CdS ($\chi_V = -1.5\times10^{-6}$ cgs units). These anomalous magnetic effects are observed at both RT and LN_2 temperatures at atmospheric pressure.

ACKNOWLEDGEMENT

The authors acknowledge the helpful discussions with Drs. Edmund Brown and Paul Cote, the ac diamagnetic measurements by John Barrett, and the technical assistance of William Yaiser. The Eagle-Picher sample material was provided through the kind offices of Dr. J. Bray of the General Electric R&D Center.

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ILLUSTRATIONS

INTRODUCTION

An anomalous, diamagnetic transition in pressure quenched cadmium sulfide, (CdS) has recently been reported by Homan and Kendall.[1] Using a low frequency technique (Hartshorn bridge) a transition from a strongly diamagnetic state to a paramagnetic state (or weakly paramagnetic to strongly paramagnetic state, etc.) was observed at temperatures up to 150K in CdS specimens that were pressure quenched at rates of about 10^6 bars/sec, from pressures just above the semiconductor-conductor transition. These specimens exhibited a layered morphology consisting of opaque, lenticular platelets of varying thickness imbedded in a compacted powder matrix.

The character of these platelets was studied by optical and scanning electron microscopy, transmission x-ray analysis, microhardness and chemical etching techniques. The platelets are mechanically and chemically different from the matrix material and are probably amorphous, although microcrystallinity cannot be ruled out. They comprise a volume fraction of 10 to 15 percent, the larger fraction being obtained by quenches from the higher pressures.

In order to obtain more quantitative measurements of the magnetic properties and to preclude the possibility of a spurious ac effect, magnetic moment measurements as a function of static magnetic field strength were performed and these results are reported in this present paper.

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[1] C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 March 1979. Details published in Benet Laboratory Technical Report No. ARLCB-TR-79-004 April 1979.

APPROACH TO THE PROBLEM

Samples were prepared as in reference 1, from optical grade CdS powder,[2] compacted into discs and then pressurized in a gasketed Bridgeman anvil pressure device loaded in a servo-hydraulic tensile testing machine. The sample resistance during pressurization was determined by measuring the resistance across the anvils using dc specimen currents not exceeding 10 milliamps. A typical resistance vs pressure trace is shown in Figure 1. The pressure values shown in this figure are approximate since an accurate pressure calibration for this device was not determined. The specimen resistance was taken to be a reproducible measure of pressure, since the sample geometry and weight were nearly identical at approximately 3.0 mm in diameter and 0.5 mm thick, and weighs 14.5 mg.

Samples were quenched from pressures which produced specimen resistance values ranging from 50 ohms to 8 ohms. Pressure quench rates were either 1 to 2 x 10^6 bars/sec, 5 to 6 x 10^6 bars/sec or less than 10^3 bars/sec.

The magnetic moment measurements were made using a Princeton Applied Research vibrating sample magnetometer. Extensive previous use has shown no evidence for experimental artifacts. However, a

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[2]Samples were prepared from optronic grade CdS from Alpha Inorganics, Stock No. 20130 compacted in a small piston cylinder device to a pressure of about 2 kbars. Starting pellets were found to be near theoretical density by a gravimetric technique. Spectrographic analysis by the spark emission method and a quantitative chemical analysis of our samples indicated the following metallic impurities: Fe = 12 ppm, Mg = 3.6 ppm, Cu = 4.2 ppm, Ag = 3.5 ppm, Bi < 1 ppm, Al < 1.4 ppm, Ge < 1 ppm, Not detected, Ni < 1 ppm, Mn < .46 ppm, Pb < .07 ppm, Cr < 2.8 ppm, Co < 1 ppm.

series of tests were also performed during these measurements to check for spurious results. These included measuring comparable signals using known materials ($CuSO_4$, $5H_2O$) and obtaining agreement with literature values of the susceptibility, changing specimen holders, changing geometrical configurations of pick-up wires, etc., and running the instrument without the cylinder normally surrounding the vibrating rod. In all cases, no evidence for deviant behavior was observed.

It had been previously found that CdS samples pressure quenched from resistance values of the order of 50 ohms at 2×10^6 bars/sec were metastable in their magnetic properties at room temperature with recovery times in the order of hours. Therefore, after pressure quenching at room temperature, these samples were kept at 77K for about an hour before being placed in the sample holder of the magnetometer at room temperature and again cooled to 77K for making the magnetic measurements.

RESULTS

The results in Figure 2 represent the average of several runs on the same sample that was pressure quenched from 50 ohms at 1 to 2×10^6 bars/sec. The result is typical of such pressure quenched samples, although some variation in the magnitude and position of the discontinuities in magnetic behavior is observed between specimens. We show for comparison the magnetic behavior of the original, unpressurized starting material, and of samples slowly pressure quenched at about 10^3 bars/sec from the same pressure. The magnetic behavior of the

empty sample holder is also shown for comparison. The small paramagnetism of the sample holder is due to absorbed oxygen.[3] The same sample holder was used during the whole sequence of measurements.

As can be seen, the magnetic behavior of the fast quenched specimens shows several striking features. First, the sample is strongly diamagnetic to around 50 Oersted, with the magnitude of the volume susceptibility being -5×10^{-5} cgs units. Above about 70 Oersted, there is a transition to an intermediate magnetic state in which the magnetic moment is positive and whose susceptibility is about $+2 \times 10^{-4}$ cgs units. Above 150 Oersted, these samples transformed to a state whose positive susceptibility is at least twice as large as the intermediate state. The calculations were made on the assumption that the entire sample is contributing to the magnetic behavior. Corrections were made for the magnetic behavior of the Teflon sample holder.

The samples were slowly warmed to room temperature in the order of several hours, during which time recovery of the magnetic behavior occurred as evidenced by a subsequent run at 77K in which these large magnetic effects were not observed. Following another short room temperature anneal, the ac diamagnetic transition previously described was observed on warming from 4.2K in the mutual inductance bridge at 20K. One further room temperature anneal lasting about an hour was made and no ac diamagnetic transition occurred above the Pb superconducting transition calibration temperature.

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[3]M. Centanni and P. A. Casabella in "Amorphous Magnetism" edited by R. A. Levy and R. Hasegaw. Plenum Press, NY, 1977, pp 663-671.

Preliminary studies on the stability of the magnetic properties indicate that slight increases in pressure and quench rate result in enhanced stability. For example, samples pressure quenched from a resistance of about 10 ohms at a rate of about 5×10^6 bars/sec are more stable in their magnetic properties, with the strong diamagnetism remaining for days rather than hours at room temperature.

The form of the magnetization curves is the same as that shown in Figure 2 and no systematic variation of the volume susceptibilities with these quenching parameters was found. However, variations in the critical fields were noted.

A sample quenched from 15 ohms at 2×10^6 bars/sec was tested for magnetic behavior at both room temperature and 77K. At both temperatures the form of the results was similar to that shown in Figure 2. However, the lower critical field (shown at 50 Oersted in Figure 2) occurred at 88 Oersted at 77K and 40 Oersted at room temperature.

Preliminary investigation of the effect of impurities was made on samples fabricated from CdS powder from a different source.[4] This sample material was lighter in color suggesting a different impurity content. Based on manufacturers data, it had a lower total impurity content. These samples showed significantly different

[4]These samples were prepared from CdS from Eagle-Picker, Ultrahigh Purity Grade. The nominal analysis provided by the manufacturer was qualitatively examined by the spark emission technique. This analysis suggested less total impurities than the original material and showed traces of Fe, Mg, Cu, Ag, Bi, Al, Ge Not detected, Ni, Mn, Pb, Cr, Co.

resistance vs pressure curves. Samples pressure quenched from a resistance of about 30 ohms at 5 to 6 x 10^6 bars/sec showed different scanning electron microscopic morphology and significantly less paramagnetic effects. This suggests that the impurity spectrum and concentration in the parts per million range may be significant and warrant further study.

DISCUSSION

We note that the magnetization curves shown in Figure 2 can be simulated by the superposition of some regions in our sample behaving with strong positive magnetic behavior (paramagnetism) with other regions behaving with classical type II superconducting magnetic behavior as shown in Figure 3. However, careful experimental determination of other physical properties, such as the resistivities of our sample or the platelets must be made as functions of temperature and magnetic field in order to either confirm or deny this intriguing possibility. Experiments to determine these additional physical properties are in progress at the present time.

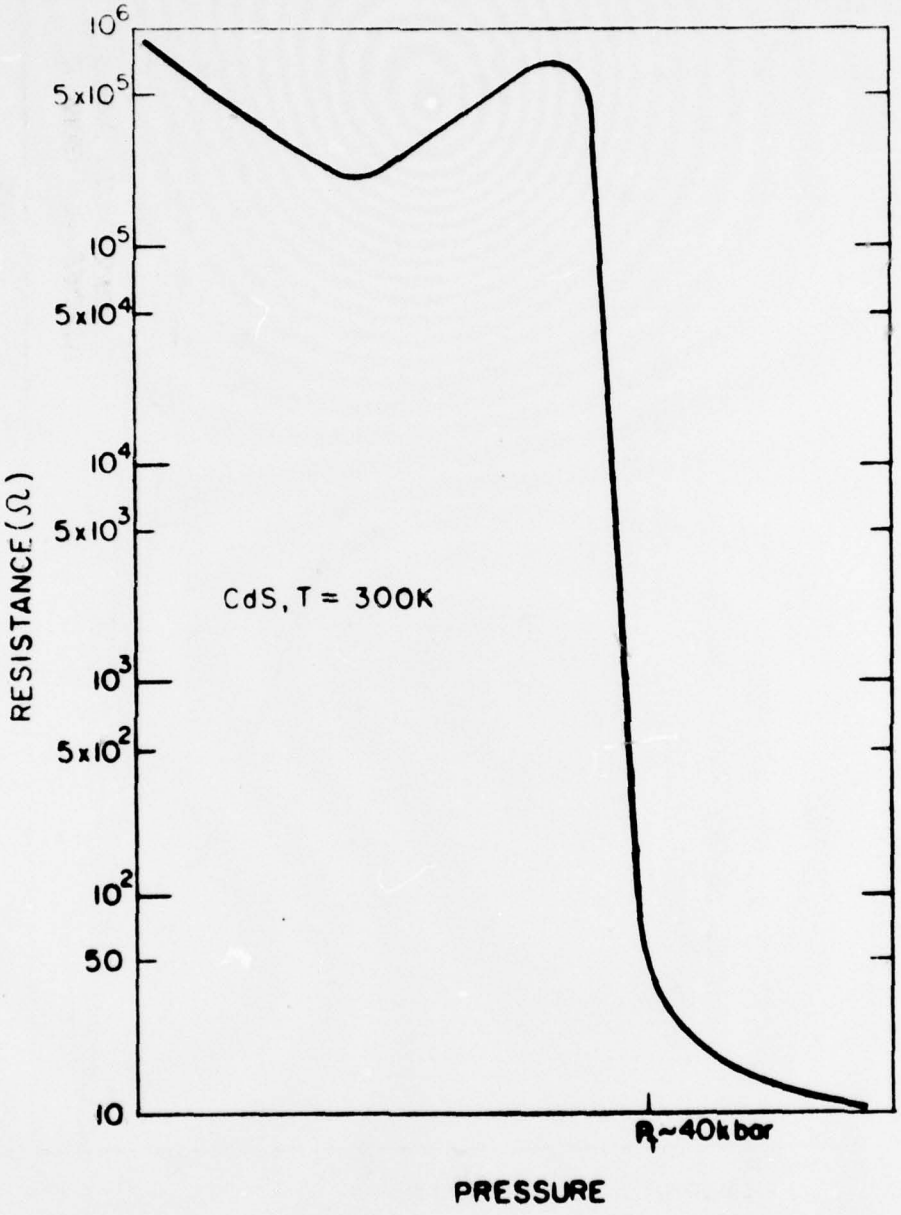

Figure 1. Typical resistometric trace of optical grade polycrystalline CdS. See ref. 2

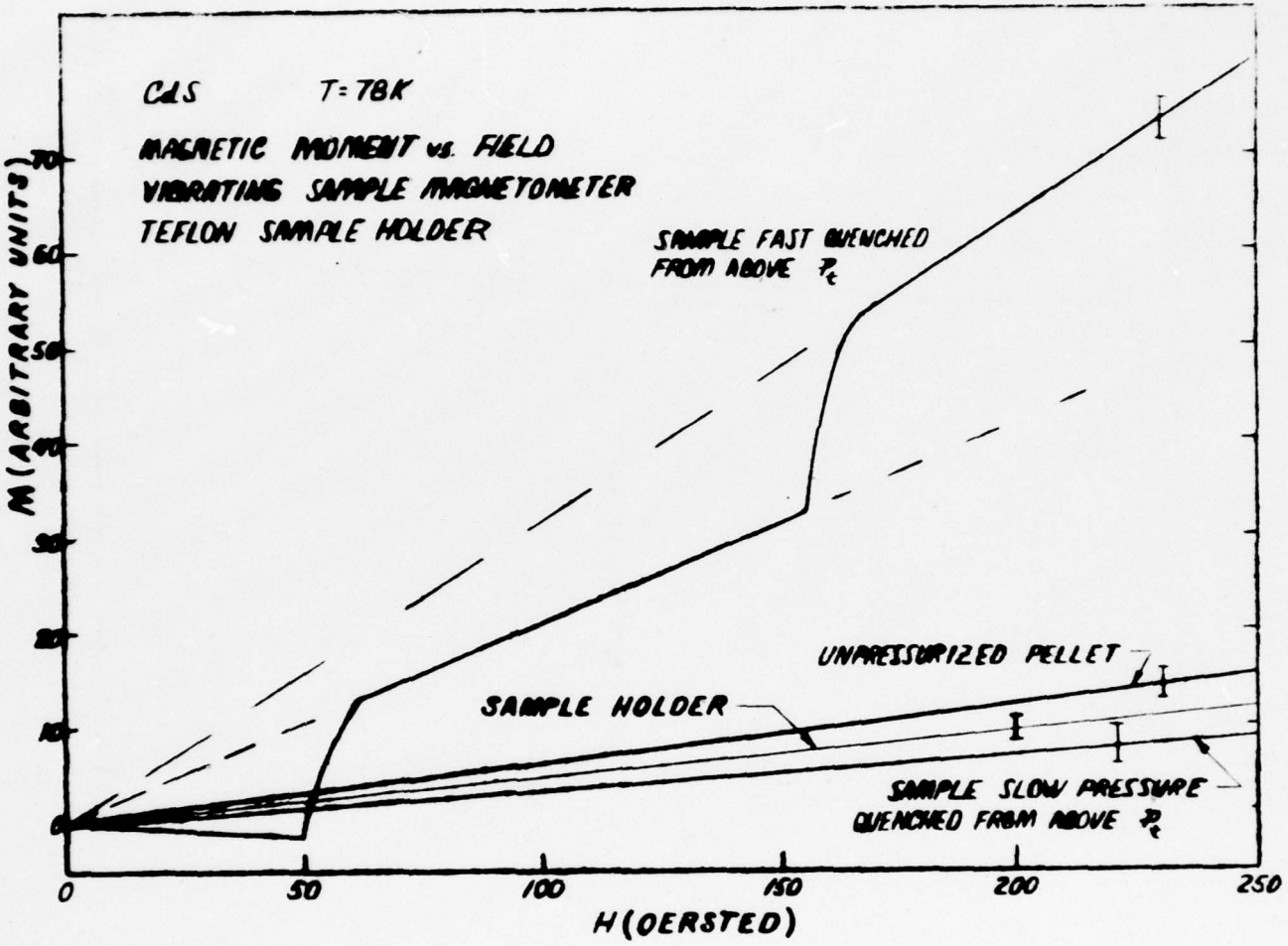

Figure 2. D.C. magnetic moment measurements vs field for optical grade polycrystalline CdS after various pressure treatments.

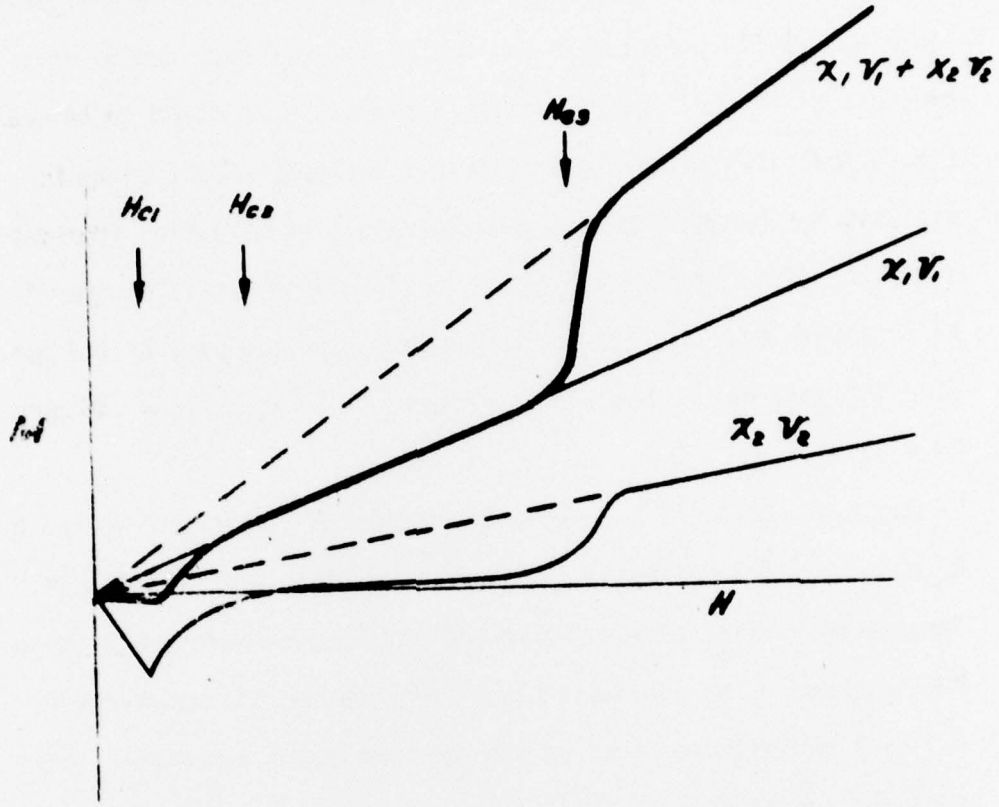

Figure 3. Simulated magnetic moment behavior of a sample containing a volume V_1 of positive magnetic susceptibility χ_1 and a volume V_2 of superconducting material assuming a linear superposition of magnetic moments.

REFERENCES

1. C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 March 1979. Details published in Benet Laboratory Technical Report No. ARLCB-TR-79-004, April 1979.
2. Samples were prepared from optronic grade CdS from Alpha Inorganics, Stock No. 20130 compacted in a small piston cylinder device to a pressure of about 2 kbars. Starting pellets were found to be near theoretical density by a gravimetric technique. Spectrographic analysis by the spark emission method and a quantitative chemical analysis of our samples indicated the following metallic impurities: Fe = 12 ppm, Mg = 3.6 ppm, Cu = 4.2 ppm, Ag = 3.5 ppm, Bi < 1 ppm, Al < 1.4 ppm, Ge < 1 ppm, Not detected, Ni < 1 ppm, Mn < .46 ppm, Pb < .07 ppm, Cr < 2.8 ppm, Co < 1 ppm.
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